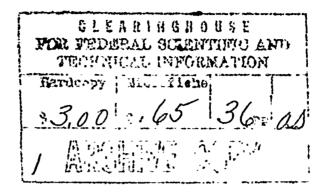
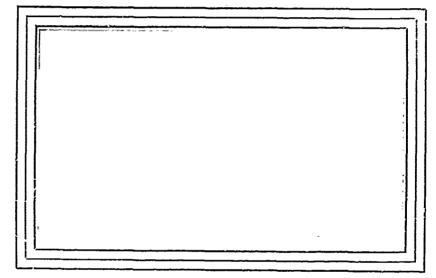
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U. S. NAVAL AMMUNITION DEPOT CRANE, INDIANA



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U. S. NAVAL AMMUNITION DEPOT Crane, Indiana

RDTR No. 51 28 Cotober 1966

BINDING PROPERTIES AND OTHER CHARACTERISTICS
OF SEVERAL POLYESTER RESIN BINDERS
USED 'IN PYROTECHNIC FORMULATIONS

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ABSTRACT

The binding strengths of pellets subjected to tensile and shear stresses, and the burning time and candlepower of flares pressed from compound aged 0-6 hours in increments of one hour prior to pressing were determined after curing periods of 5 and 30 days. Three formulations were used, each containing the same ratio of magnesium/sodium nitrate/binder; but, with three different polyester resin binders—Laminac 4110.

Laminac 4116, and Aropel 7720M.

The variation in physical strength, candlepower, and burning time with respect to delay time between mixing and pressing was found to be greatest for pellets and candles containing Laminac 4110. Likewise, the physical strength of pellets containing Laminac 4110 was significantly higher than the other pellets after curing 5 days; however, tests after 30 days indicated that by this time the Laminac 4116 had essentially fully cured, and now had binding properties similar to Laminac 4110. The physical strength of pellets cured at 75 - 85°F with Aropol 7720M was considerably less then pellets containing the other binders.

All units cured for 16 hours et 150°F, and then tested efter 5 days exhibited considerably higher strengths than

pellets not subjected to an elevated temperature. However, tests after 30 days showed that for the Leminac binders, greater binding strength is obtained by curing at room temperature for the duration of the curing cycle.

TABLE OF CONTENTS

		Page
Objecti	LV8	1
	mental Nethod	1
-	position Formulation	1
Pre	essing and Curing Procedure	1
	sting Apparatus and Equipment	2
	sion	3
	iders	3
	sile Strength	5
	ear Strength	7
	fect of 150°F Curing Cycle	9
	rning Time and Candlepower of Flares	10
	llet Density	11
	sions	12
0 0		
ILLUST	RATIONS A, B & C	4
TABLES		
I	Typsile Test Experimental Data	14
II	As " ge Failure Loads (Lb.) in Tonsile	15
ILE	r Test Experimental Data	16
IV	Av age Feilure Loads (Lb.) in Shear	17
V	Experimental Data - Pellets Cured at 150°F	18
VI	Average Tensile and Shear Feilure Loads (Lb.)	
	or Pellets Cured a 150°F and Ambiently	19
VII	h'yerimental Data - Burning Time & Average	
	Candlepower of Candles	20
VIII	Burning Time, Candlepower & Candlepower-Seconds	
	of Flares	21
CAMPHS		
I	.,,	22
II	.,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	23
III		24
IV	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	≲5
v		26
٧Ï		27

I. OBJECTIVE

The purpose of this study was to determine the binding properties of several polyester resins used in pyrotechnic compositions as a function of the polymerization time before and after pressing. Additionally, the variation in cardle-power and burning time of the formulation under consideration was to be investigated.

II. EXPERIMENTAL METHOD

Composition Formulation

A pyrotechnic formulation containing 61.4% magnesium (granulation 18), 33.8% sodium nitrate (Class 2), and 4.8% binder was selected as the composition to be investigated. Three different polyester binders were utilized—Laminac 4110 and 4116 (American Cyanamid Co., Wallingford, Conn.), and Aropol 7720M (Archer-Daniels Midland Co., Minneapolis, Minn.). After mixing, the compound was stored in a closed container until pressed.

Pressing and Curing Procedure

Pellets one inch in diameter and two inches long were pressed immediately after mixing, and in increments of one hour after mixing up to a maximum of six hours. Each pellet contained two increments of 22 grams each, pressed at a dead

load of 8,000 pounds. Twenty-four pellets were pressed immediately after mixing, and 12 during each time interval thereafter. The diameter, length, and weight of each pellet was recorded immediately after pressing, and in some cases after five and 30 days curing. All pellets were cured in a sealed can at 75 ~ 85°F, except for 12 pellets pressed immediately after mixing which were cured for approximately 16 hours at 150°F. The pellets were tested five and 30 days after pressing.

Three flares were pressed immediately after mixing, and during each hour thereafter, up to six hours after mixing. Each flare contained three 200 gram increments, pressed at a deadload of 22,000 pounds into a fish paper tube which was 1.96" OD and 1.75" ID. The flares were cured at 75 - 85°F, and burned after 30 days.

Testing Apparatus and Equipment

The testing apparatus and equipment used in the determination of the shear and tensile binding properties of the various resins are illustrated on the following page. The testing machine used to perform both the tensile and shear tests is shown in Illustration A. The movable jaws were clamped together to hold the cables which were connected to

apart to supply the required force. The shear and tensile test fixtures are shown in Illustrations B and C respectively. For the shear test, the pellet was placed in the test fixture and then the assembly was mounted in the test machine. In the tensile test, a pellet with an end plate attached to each end was held in the fixture by the removable pin, and then the assembly was placed in the testing machine.

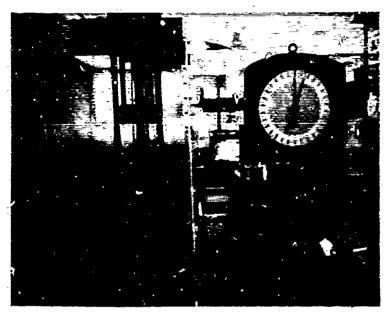
Orbond 121 was used to bond the end plates required for the tensile test to the pellets. The tensile test data for the Leminac 4116 units is rether incomplete because an inadequate amount of adhesive was used to bond the end plate to the pellet; thus, failure occurred at the plate-pellet interface rather than at the increment.

IV. DISCUSSION

Binders

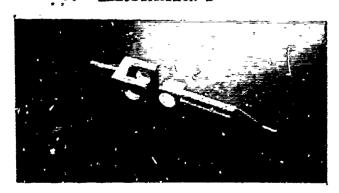
The binders selected for this investigation were ones believed to be suitable for use in pyrotechnic flare compositions, and which would essentially fully cure at temperatures between 75 - 85°F when adequately catalyzed. Cobalt Naphthenate, which promotes room temperature cure when catalyzed by methyl ethyl ketone (Lupersol DDM), is present in a

ILLUSTRATION A



Riehle Testing Machine
Mfg. by American Machine & Metals Co.
East Moline, Illinois

ILLUSTRATION B



Shear Testing Fixture

ILLUSTRATION C



Tensile Testing Fixture with end plate

at room temperature. On the other hand, Leminac 4116 contains a lesser amount of Gobalt Naphthenate; therefore, this resin will not cure as quickly as Laminac 4110, and in some cases an elevated temperature may be required. The Aropol resin does not contain any of the Gobalt promoter, hence, the promoter must be added to obtain a room temperature cure.

Company literature indicates that the gel time for Laminace 4110 and 4116 with 1.5% impersol DDM catalyst, is approximately 15 minutes and 35 minutes respectively, and that Aropol 7720M should gel in about 25 minutes when catalyzed by 0.5% Cobalt Napthenate and 1.0% impersol DDM. Although the composition was stored in closed containers between mixing and pressing, it would be suspected that the influence of the delay time between mixing and prascing upon the physical properties and burning characteristics of the pressed composition would be most noticeable in the laminace 4110 composition. The literature also states that the tensile strength of the fully cured laminace resins is approximately 9,000 PSI, and about 2,500 PSI for the Aropol binder.

Tensile Strength

Graph I shows a binder-binder comparison between the

days ouring, while Graph II illustrates the difference in tensile strength after five and 30 days for a given binder. As stated previously, the experimental data for the Laminac 4116 units is incomplete because the end plate-pellet bond proved to be weaker, in many cases, than the increment-to-increment bond. All of the data considered was for failures which occurred at the increment-to-increment junction. Table I gives the experimental tensile failure loads after five and 30 days for the various mix-press delay times, and Table II contains the average of the above figures for a given resin and delay time.

Considering the accuracy of the testing equipment used, and the variation in the data for a given binder and delay time, the delay time between mixing and pressing does not appear to have a significant effect upon tensile strength.

The tensile strength of the Aropol pellets did seem to exhibit somewhat of a trend toward increasing as the delay time increased; however, the magnitude of the increase can hardly be judged significant. A binder-binder comparison shows that the Laminac 4110 pellets sustained a 40% greater tensile force before failure than the Laminac 4116 units, and 80% greater

than the Aropol pellets.

n the other hand, the difference in tensile strength after five and 30 days ambient cure for a given binder was found to be very significant. The Laminac 4110 units failed at a 65% greater tensile load after 30 days than five days, the Laminac 4116 pellets increased 100% in tensile strength, and the Aropol pellets exhibited an increase of 50%. again, the Laminac 4110 pellets were superior to the other units, since their tensile strength was 10% and 80% greater, respectively, than the Laminac 4116 and Aropol 7720M pellets. It is interesting to note that the tensile strength of the pellets increased substantially between the five and 30 day period, and that efter 30 days cure, the Laminac 4110 and 4116 pellets exhibited scape hat camerable tensile binding properties. This data agrees with the company literature for the various binders, which indicated that Laminac 4116 does not cure as quickly as 4110, and that when fully cured, the tensile strength of the two Leminac resins are basically the same, and considerably greater than for, Aropol 7720M.

Shear Strength

The shear strength data is presented in Tables III and IV. Graphs III and IV give a binder-binder comparison of

shear strengths after five and 30 day curing cycles, respectively and Graph V shows the variation in the shear strength for a given binder, resulting from a five and 30 day cure.

Referring to Graph V, it is seen that as the time from mixing to pressing increases from 0 to 6 hours, the shear strengths of the Leminac 4110 units increased rather consistently, amounting to an overall increase of approximately 200%. Such a trend was not evident after the 30 day cure in the Leminac 4110 units, nor did it exist after either five or 30 days for the other binders.

0

After five days cure, the pellets containing Leminac 4110 failed at about a 50% greater shear load than the other two pellet formulations. However, tests after 30 days revealed that although the Leminac 4110 had a shear strength approximately 200% greater than the Aropol units, they now failed at a 25% lower shear load than the Leminac 4116 pellets. This data substantiates that found during the tensile tests, in that although the physical properties of Laminac 4116 are inferior to Laminac 4110 after a short ambient curing cycle, as the curing period is increased, the physical properties of the Laminac 4116 are enhanced until the two binders have basically the same properties. The per cent increase in the

shear strengths after five and 30 days was 125%, 350%, and 15% for the Laminac 4110, Laminac 4116, and Aropol resins, respectively.

Effect of 150°F Curing Cycle

Table V contains the experimental data for pellets cured approximately 16 hours at 150°F, immediately after pressing, and then stored in closed containers at 75 - 85°F until tested after five and 30 days. The average values for the above data are given in Table VI, along with the corresponding values of pellets cured at room temperature.

If the tensile and shear strengths of pellets subjected to the elevated temperature are compared to those cured at room temperature, it is seen that the elevated temperature cure has a marked effect upon the physical properties of the resins. After the 150°F cure, the Aropol units tested after five and 30 days failed at tensile and shear loads which were at least 75% and 20% greater, respectively, than the Leminac resin pellets. Referring to Table VI it is seen that the Leminac pellets cured at an elevated temperature and then tested after five days, possessed significantly greater tensile and shear strengths than pellets not subjected to the 150°F cure; however, the results of similar units tested after 30

days indicated that binding strengths of units oured solely at 75 - 85°F, were approximately 25% greater than those cured for 16 hours at 150°F and the remaining time at 75 - 85°F.

Burning Time and Candlepower of Flares

There was found to be a significant variation in candlepower, burning time, and the emitted candle-power-seconds with
respect to the delay time between mixing and pressing. A
similar difference also existed between the three binders.

For the flares containing Laminac 4110, 4116, and Arcpol 7720M,
an overall decrease of 6%, 5%, and 3% in burning time, an
increase of 14%, 8%, and 8% in candlepower, and an increase
of 7%, 4%, and 4% respectively in candlepower-seconds were
obtained for compound pressed between 0 - 6 hours after mixing. This experimental data may be found in Table VII.

Average values are given in Table VIII and plotted on Graph VI.
Generally consistent data was obtained for a given delay time
and formulation.

If an average value of the emitted candle power-seconds is calculated, it is found that the Arcpol and Laminac 4110 units are approximately equal, while the Laminac 4116 flares emitted about 13% fewer candle power-seconds. The pressed length of flares of a given formulation were essentially the

same; therefore, the variation in burning time is indicative of the variation in burning rate existing for the various units.

Pellet Density

No significant difference existed between the density or weight of pellets containing a given binder for the various delay times, nor between the pellets containing the three binders. Likewise, no significant change in density occurred after curing for five and 30 days. The density of the pellets was approximately 1.700 gm/cc, with less than a 3½ variation between the units containing the three resins.

V. SUMMARY

Of the three resins investigated, the optimum binder for use at room temperature curing conditions appears to be Laminac 4110, when it is considered that the function of a binder is to supply physical strength to the pressed composition while at the same time impart a minimum amount of degradation to the perfermence of the flare. The burning performence of the Aropol flares compared favorably with the Laminac 4110 units; however, the relatively poor physical strength properties of the Aropol pellets after both five and 30 days curing, make this resin inferior to Laminac 4110.

A comparison between the two Laminac resins indicates that after an extended curing period, the two compare favorably in physical strength; however, after only five days ambient cure, the Laminac 4110 units possessed superior strength properties. Also, the number of candle power-seconds emitted by the Laminac 4110 flares was about 12% greater than for the 4116 units.

The results of this study also indicate that a short elevated temperature cure will substantially increase the tensile and shear strength of the compositions investigated, compared to a short ambient cure. However, for the Laminac resins, it appears that units cured 30 days ambiently have significantly higher physical strengths than units subjected to a 16 hour 150°F cure and then cured ambiently for the remaining 29 days. Theoretically, the Aropol resin should have cured at 75 - 85°F; however, considering the physical strength data after the 150°F cure, it is evident that this resin did not cure substantially at room temperature.

VI. CONCLUSIONS

1. Laminac 4110 was found to be a superior binder compared to Laminac 4116 and Aropol 7720M, considering both physical strength and burning parformance of pellets and flares, respectively.

- 2. Aropol 7720M exhibited poor binding properties when cured at room temperature but compared favorably with the Leminac resins when cured at 150°F for 16 hours.
- 3. Of the three resins investigated, Laminac 4110 will impart superior tensile and shear strength to the pressed composition after several days ambient oure; however, after approximately 30 days the two Laminac resins possess essentially the same physical strengths.
- 4. Flares containing the Arcpol 7720M and Laminac 4110 resins emitted a significantly greater number of candlepower-seconds than the units containing Laminac 4116.
- 5. The burning time and candle power of flares decreased and increased respectively, a significant amount, as the delay time between mixing and pressing increased.

TABLE I

TENSTLE TEST Laminac 4110 5 Day 30 Day - 315 lbs. 173 lbs. 365 lbs. 185 lbs. 285 lbs. 180 lbs. 285 lbs. 198 285 " 296 " 297 " 297 " 297 " 298 189 285 285 189 285 285 285 285 285 285 285 285 285 285		*	Day	Olbs.	=		O lbs.		0	Olbs.		0	O lbs.	= 10	= 0	0 lbs.		2 "	7	= 0	5 "	0 lbs.		ء د	
Laminac 4110 Laminac 4116 5 5 5 5 5 5 5 5 5		01 7720	30	• 16	76	15	•		16	•		15	•		1.6	•		17	•		17			17	-
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TENSTLE TEST Laminac 4110 5 Day 30 Day - 315 lbs. 173 lbs. 365 lbs. 185 lbs. 285 lbs. 180 lbs. 285 lbs. 198 285 " 296 " 297 " 297 " 297 " 298 189 285 285 189 285 285 285 285 285 285 285 285 285 285	ral data	4116	30 Day			2	300 lbs.	ı	1	ı		3	1	ı		1	1	1	ı	ı	1	1	1	ī	
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TABLE II

TABLE III

- O CLARATE THE PROPERTY OF STREET, ST		SHEAR TE	SHEAR TEST EXPERIMENTAL DATA	TAL DATA		-
Mix-Pross		4110	Laminao	4116	Aropol 7	7720M
Dolay Timo	5 Day	30 Day	5 Day	30 Day	!!	30 Day
	398 189					2
	· ()		יהסיד ססס	TOOT TOOT	00% 10g.	475 lbs.
ن -		0220 T 2220				485 "
Appendix to be block to the control of the control	3	765	782	1665 "	405 "	480 "
,	465 1bs.	1655 lbs.	362 lbs.	2035 lbs.		•
ri 			382 "		370 "	425 "
		1,500 "	380 "	1625 "	365 "	445 "
	555 108.	1525 lbs.	545 1bg.	1755 lbs.	455 lbs.	525 1hg.
N 3						500 "
efective in: Total 21 pt print the section in the s	522 =	1300 "	540 "	1625 "	460 "	515 "
1	665 1bs.	1425 lbs.	444 lbs.	ł .	475 lbs.	
 			484 "	1905 "		550 "
· Out I control as on a spiritual instruction designs to be a second	3	1			527 "	" Ors
•	825 155.	1615 lbs.	488 1bs.	1990 lbs.	ı	•
₹					465 "	545 "
And the of the statement of the statemen	773 =	1425 "		1740 "	427 "	540 "
•	613 lbs.	1435 lbs.	350 lbs.	2025 lbs.	429 1bs.	
ລ	78					510 "
No. 14 of the state of the stat	į		377 "	2015 "	462 "	210 "
,	358 1bg.	1500 1bs.	427 lbs.	2035 lbs.	475 lbs.	580 lbs.
9						555 "
	: 000T	129%	426 "	# 00T2	520 "	545 "
Countesparation of the second contest of the second of the	The property of the American			,		

TABLE IV AVERAGE FAILURE LOADS (LB.) IN SHEAR

Bindor Luminac 4110 Luminac 4116 Aropol 7720M Curing Foriod 5 Day 30 Day 5 Day 5 Day 5 Day 30 Day Time between mixing and pross- 430 1595 305 1815 383 480 1ng 650 1565 375 1885 355 420 1
--

TABLE V

EXPERIMENTAL DATA - PELLETS CURED AT 150°F

	5 De	y	30 D	ау
Binder	Tensile	Shear	Tensile	Shear
Laminac 4110	215 lbs. 250 "	1308 lbs. 1425 " 1500 "	210 1bs. 180 "	1230 lbs. 1275 " 1180 "
Laminac 4116	234 lbs. 184 "	1375 lbs. 1215 " 1460 "	260 lbs.	1455 lbs. 1395 " 1475 "
Aropol 7720M	360 lbs. 427 " 412 "	1690 lbs. 1870 " 1880 "	570 lbs. 510 " 510 "	1740 lbs. 1705 " 1665 "

TABLE VI

AVERAGE TENSILE AND SHEAR FAILURE LOADS (LB.) FOR PELLETS CURED @ 150°F AND AMBIENTLY

	***************************************	,	i .	:		Andrew II .			
			SASO S				30	30 Days	,
Binder		T	Tensile				116		Shear
		150°	Ambient	150	150° Ambient	150°	Ambient	150	150° Ambient
Lemine	Leminec 4110	230	180	1410	670	195	300	1230	1510
Lamine	Lominac 4116	210	130	1350	420	260	265	1440	1900
Aropol	Aropol 7720M	400	011	1815	450	530	165	1705	515

TABLE VII

EXPERIMENTAL DATA - BURNING TIME & AVERAGE CANDLEPOWER OF CANDLES

	-				-		-	_			~~~	****			_			•					→
7720M	G.P.	209,000	216,000	217,000	222,000	226,000	217,000	221,000	224,000	213,000	215,000	230,000	217,000	224,000	227,000	231,000	223,000	_	227,000	226,000	228,000	237,000	
Aropol 77	B.T.	140 Sec.		140 "	136 "	139 "	139 "	138 "	135 "	140 "	137 "	138 "	137 "	141 "	134 "	135 "	1.32 "	137 "	136 "	134 "	136 "	137 "	
4116	C.F.	193,000	212,000	187,000	212,000	198,000	197,000	201,000	200,000	181,000	201,000	206,000	221,000	205,000	209,000	210,000	198,000	201,000	246,000	220,000	218,000	200,000	
Laminac 41	B.T.	Soc.	=	134 "	=	=	=	=	=	 =	- -	=	=	=	=	=	=	=	=	129 "	188 "	127 "	
4110	C.P.	212,000	223,000	226,000	219,000	245,000	232,000	232,000	242,000	237,000	225,000	235,000	240,000	233,000	231,000	245,000	245,000	247,000	242,000	254,000	247,000	252,000	
Leminac 4	B.T.	131 Sec.		129 "	129 "	129 "	129 "	124 "	128 "	128 "	128 "	92°C	127 "	126 "	126 "	- 555	124 "	124 "	123 "	120 "	126 "	. ogt	
Mix-Press	Delay Time		0 Hrs.			<u>-</u>			α			63			4			S			9		

B.T. - Burning Time C.P. - Average Candlepower

RDTR No. 51

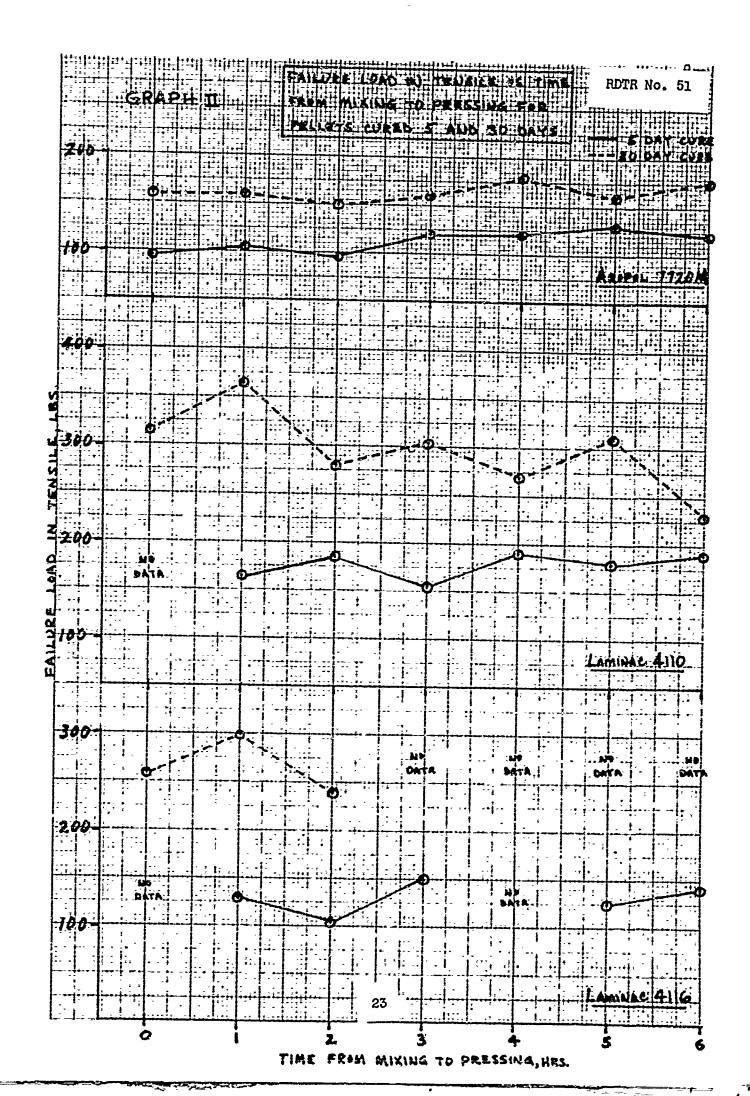
TABLE VIII

BURNING TIME, CANDLEPOWER AND CANDLEPOWER-SECONDS OF FLARES

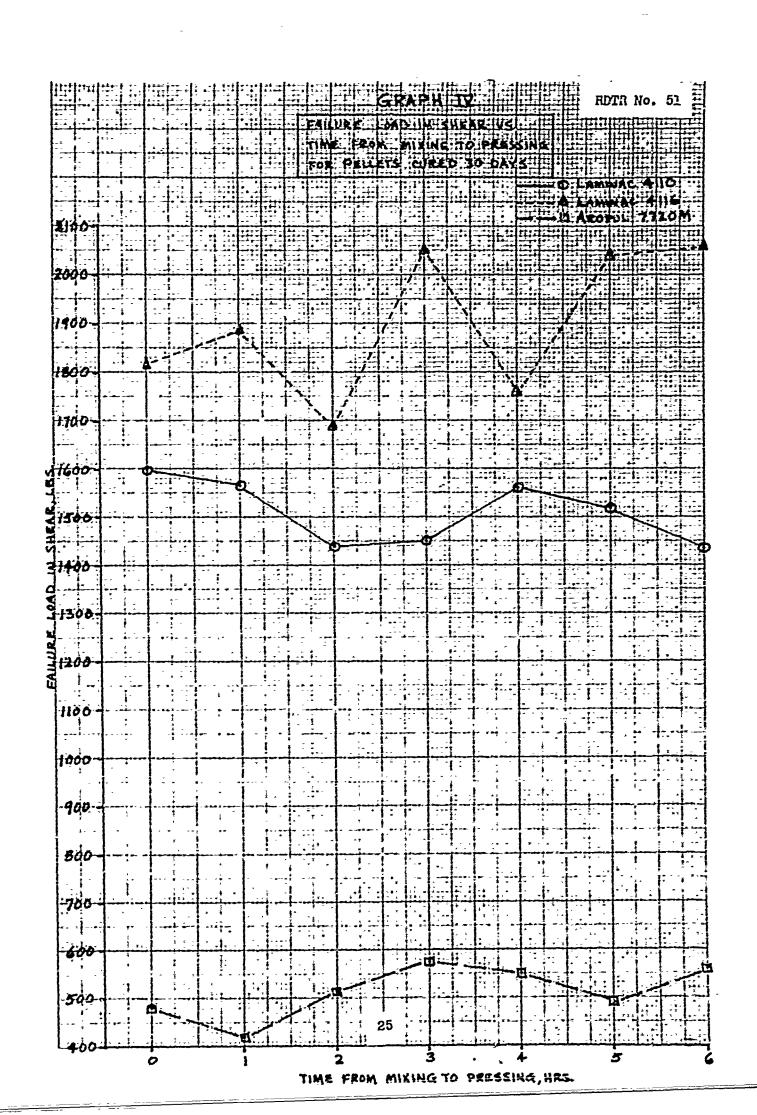
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mixing end		Leminac 4110	.10		Laminac 4116	116		Aropol 7720M	MO
pressing	B.T.	C.P.	C.PSec	B.T.	G• P•	G.PSec	B.T.	G.P.	C.FSec
Ó	130	220,000	28.6 x 10 ⁶	133	000,701	26.2 x 10 ⁶	140	214,000	30.0 x 10 ⁶
Н	129	232,000	29.9	132	202,000	26.7	138	222,000	30.6
જ	127	237,000	30.1	129	194,000	25.1	136	219,000	30.2
ю	126	233,000	29.4	129	209,000	27.0	137	221,000	30.3
4	126	236,000	29.8	121	203,000	27.2	137	227,000	31.1
ເດ	124	243,000	30.1	127	215,000	27.3	135	227,000	30.6
v	182	251,000	30.6	126	213,000	86,8	136	230,000	31.3
Average	126	236,000	29.8 x 10 ⁶	130	205,000	26.6 x 10 ⁶	137	223,000	30.5 x 10 ⁶

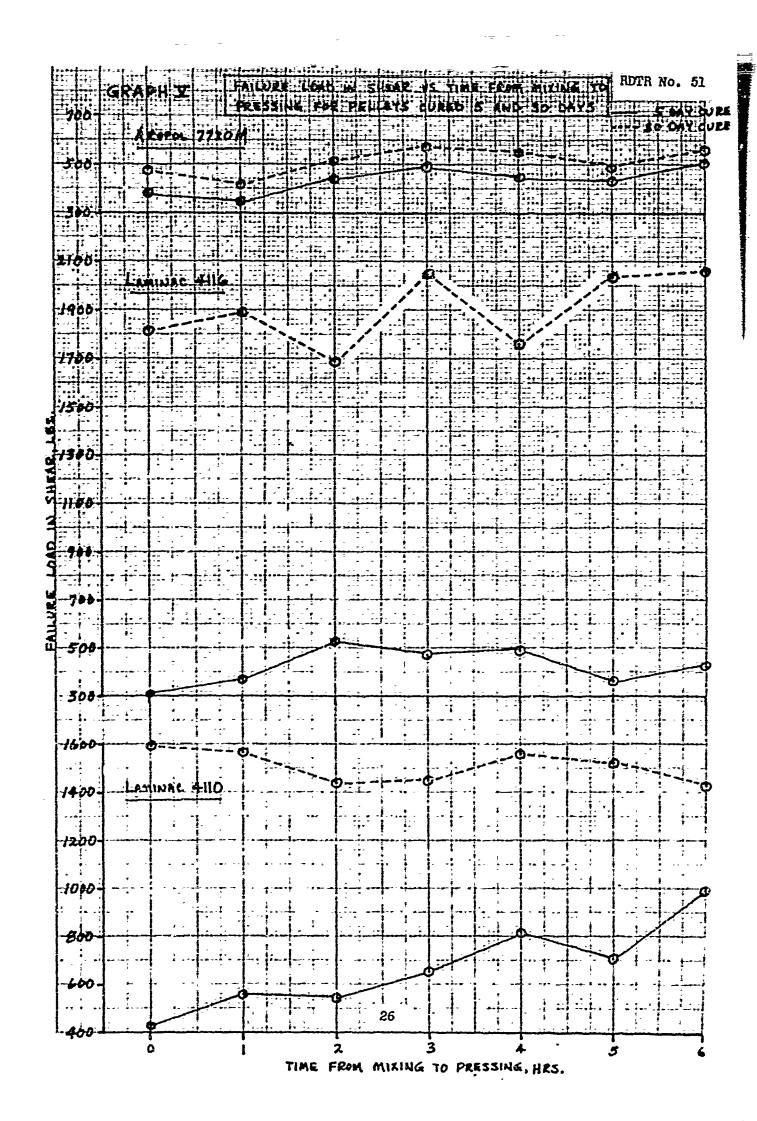
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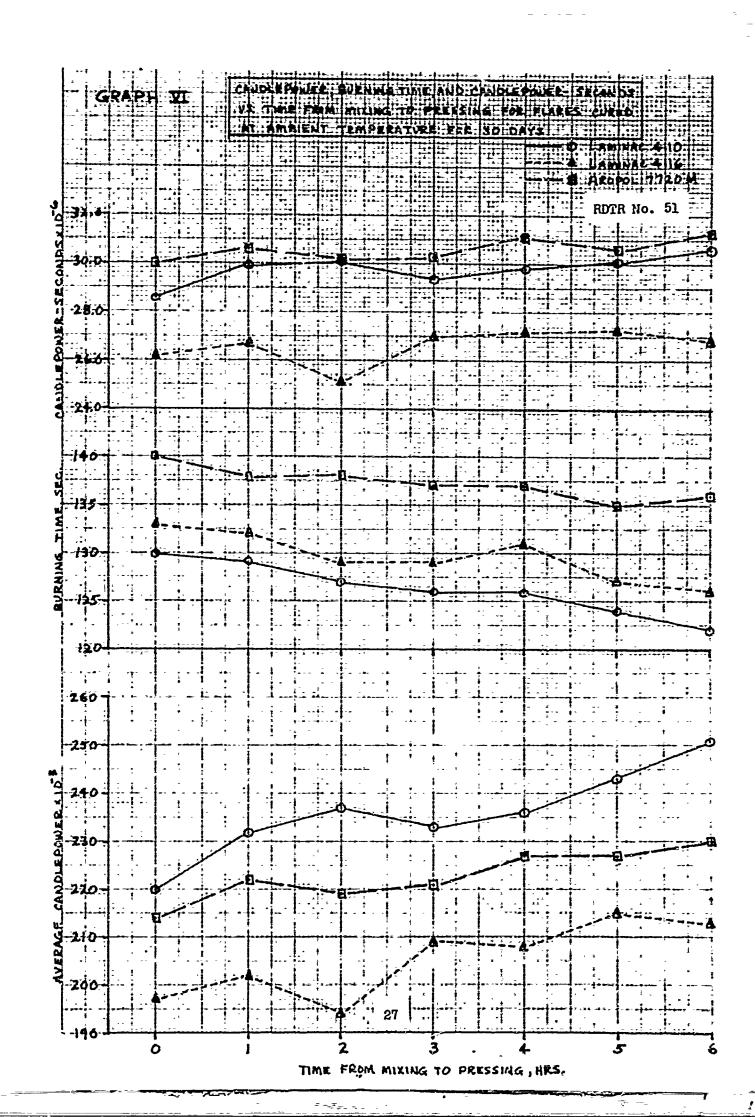
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· and	Crane, Indi	ene.	

and the burning time and candlepower of flares pressed from compound aged 0-6 hours in increments of one hour prior to pressing were determined after curing periods of 5 and 30 days. Three formulations were used, each containing the same ratio of magnesium/sodium nitrate/binder; but, with three different polyester resin binders—Leminac 4110, Leminac 4116, and Aropol 7720%.

The variations in physical strength, pendlepower, and burning time with respect to delay time between mixing and pressing were found to be greatest for pellots and candles containing Leminac 4110. Likewise, the physical strength of pellets containing Leminac 4110 was significantly higher than the other pellet after curing 5 days; however, tests after 30 days indicated that by this time the Leminac 4115 had essentially fully cured, and now had binding properties similar to Leminac 4110. The physical strength of pellets cured at 75 - 85°P with Aropol 7/20M was considerably less that pellets containing the other binders.

All units cured for 16 hours at 150°F, and then tested after 5 days exhibited considerably higher strengths than pellets not subjected to an elevated temperature. However, tests after 30 days showed that for the Laminec binders, greater binding strength is obtained by curing at room temperature for the duration of the curing cycle.

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4.	KEY WORDS	LINK A		LINK B		LINK C	
		ROLE	WT	ROLE	WT	ROLE	WT
1,	Flares, Physical Strength						
2.	Flares, Burning Characteristics]		l			
3.	Flares, Binders						
4.	Flares, Aging						
5.	Flare Composition, Aging			į			
6,	Binders, Physical Properties						
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